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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
VINCENZO CALEMMMA, ET AL. : EXAMINER: HAILEY, PATRICIA L.
SERIAL NO: 10/563,209 :
FILED: MAY 25, 2006 : GROUP ART UNIT: 1793
FOR: CATALYTICALLY ACTIVE :
AMORPHOUS POROUS SOLID AND
PROCESS FOR ITS PREPARATION

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

I, Vincenzo Calemma, a citizen of Italy, hereby declare
and state that:

1. I have a degree in Industrial Chemistry,
which was conferred upon me in 1981 by the University of Turin located in
Turin.

2. I have been employed by ENI since 06-6-1982 and I
have a total of 10 years of work and research experience in the field of
catalysis, testing, and hydrocarbon processing.

3. The following experiment was carried out by me or under my direct supervision
and control.

4. The experiment studied the effect of changing the atomic ratio Si/Al of catalytically active amorphous porous solids containing Si, Al and P from Si/Al=10 to Si/Al=50 in a process of hydrocracking linear paraffins. The Si/Al=50 resulted in production of a significantly larger amount of highly isomerized high boiling point residue, such as can be used as LUBE base without any further treatment or with a slight dewaxing treatment, further to obtaining, with good yields, middle distillates with good cold properties.

The comparative experiment was carried out according to the following steps:

1) A catalytic composition was prepared, and successively a hydrocracking catalyst, exactly repeating in sequence the procedures reported in the Examples 1, 6 and 11 of the present application, with the only difference being that – in the initial step of hydrolysis and gellification, a quantity (about 20g) of "aluminium isopropoxide" was used to obtain a ratio Si/Al =10, instead of 50. The resulting comparative catalyst (Comp C) had the following characteristics:

Table 1

Catalyst	Si/Al	Si/P	P/Al	S _{BET} (m ² /g)	V _p (ml/g)	d _{DFT} (nm)	Pt (% b.w.)
Comp C	10	50	0.2	360	0.64	10	0.3
Ex. 11	50	50	1	490	0.84	7.3	0.3

Table 1 also includes the characteristics of the catalyst obtained according to Example 11 of the present application (see specification at page 36, Table 3 for the abbreviations).

It should be noticed that, in order to maintain unchanged the quantity of phosphor used in the Example 1 of the present application, the ratio P/Al reduces to 0.2. An additional sample where the ratio P/Al was maintained equal to 1 resulted in a solid rather deprived of catalytic activity (collapse of the porous structure).

2) Each of the above catalysts was used for carrying out two tests of hydrocracking, at different conversion, according to the procedure of Example 16 of the present application.

The n-paraffinic (wax) mixture under treatment had the following composition:

Table 2

Fraction	Distillation Range (°C)	Composition % b.w.
C2-C9	< 150	0
C10-C14	150 – 260	7.35
C15-C22	260 – 370	23.77
C22+	> 370	68.88

At the end of the hydrocracking step, after fractioning, the high boiling fraction (C22+) was subjected to cold dewaxing, as described in the specification at page 39, lines 12-25, with the objective of determining the amounts of Lube base (Lube yield). The results are reported in following Table 3.

Table 3

Run No.	Catalyst	Temp. (°C)	WHSV	Conv. C22+	C1-C4	C5-C9	C10-C14	C15-C22	C22+	Lube Yield (%)	Viscosity 370+
1	COMP C	350	1.5	74.85	7.36	17.94	24.61	32.72	17.36	63	3.07
2	COMP C	353	1.5	85.77	8.55	21.72	28.78	31.13	9.82	72	2.97
3	Ex. 11	335	1.5	72.86	1.53	8.99	25.03	45.56	18.89	87	4.56
4	Ex. 11	338	1.5	86.14	1.78	10.40	29.36	48.82	9.64	93	4.35

It should be observed that the tests were carried out at a constant space velocity (same residence time). Tests 1 and 3 were carried out at middle conversion (~ 73-75%), while tests 2 and 4 were carried out at high conversion (~ 85-86%).

By a comparison between test 1 and test 2, or test 2 and test 4, it may be observed:

i. The Lube base yield (fraction C22+ isomerized) increases over 20% through the catalyst of the present invention (Si/Al = 50), and in the test 4, with a Lube yield of 93%, the high boiling residue could be used as such as a lubricating base. The improved quality of the high boiling product obtained through the catalyst of the present invention is confirmed by

the higher levels of viscosity, which reduces the addition of Viscosity Improvers (VII) in the formula of the lubricant.

ii. The comparative catalyst (COMP C) ($\text{Si}/\text{Al} = 10$) produces moreover a high quantity of gas (fraction C1-C4) and naphtha (fraction C5-C9), which are undesired products, reducing the yield in Middle Distillates with respect to the catalyst of the present invention.

The obtained results clearly show that a catalyst containing a quantity of aluminum higher than that as claimed (i.e., an atomic ratio Si/Al lower than "an atomic ratio Si/Al ranging from 20 to 250") may significantly change the catalytic properties.

In particular, it has been observed that in the presence of a ratio $\text{Si}/\text{Al} = 10$ it is not possible to obtain the desired isomerization level of the high boiling residue, while this is possible through the catalyst according to the present invention, wherein the ratio Si/Al can be, e.g., equal to 50.

5. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

6. Further declarant saith not.

Date: ____12th____, December, 2008 _____ San Donato Milanese _____



Vincenzo Calemme